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IS 4129 (1967): Methods of chemical analysis of aluminium trifluoride [MTD 13: Ores and Raw Materials]

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IS:4129-1967

Indian Standard
METHODS OF
CHEMICAL ANALYSIS OF
ALUMINIUM TRIFLUORIDE

REAFFIRMED

1966

UDC 549.455.2:543



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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 1

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TO

IS : 4129 - 1967 METHODS OF CHEMICAL ANALYSIS OF ALUMINIUM TRIFLUORIDE

(*Page 13, clause 8.4.2*) — Add the following new clauses after **8.4.2**:

9. DETERMINATION OF LOSS ON IGNITION

9.1 Reagents

9.1.1 Magnesium Perchlorate (Anhydrous)

9.2 Procedure

9.2.1 Weigh 1.00 g of sample into a previously weighed platinum crucible. Heat slowly first and ignite in a furnace at a temperature 550°C for half an hour. Take it out, cool in a desiccator containing preferably magnesium perchlorate and weigh to constant weight.

9.3 Calculation

$$\text{Loss on ignition} = \frac{A - B}{C} \times 100$$

where

A = mass of crucible with sample,

B = mass of crucible with residue after ignition, and

C = mass of sample taken.

10. DETERMINATION OF SULPHATE (AS SO₃) BY GRAVIMETRIC METHOD

10.1 Outline of the Method — The sample is fused with sodium carbonate and boric acid. The sulphate is precipitated by barium chloride, ignited and weighed.

10.2 Reagents

10.2.1 Sodium Carbonate Anhydrous

10.2.2 Boric Acid

10.2.3 Concentrated Hydrochloric Acid — (rd = 1.16) (Conforming to IS : 265-1976*).

*Specification for hydrochloric acid (second revision).

10.2.4 Dilute Ammonia Solution — 1 : 1 (v/v).

10.2.5 Methyl Red Indicator Solution — 0·1 percent in methanol.

10.3 Procedure

10.3.1 Weigh 1·00 g of sample and mix with 4 g of sodium carbonate and 2 g of boric acid in a platinum crucible. Fuse over a burner or in a muffle furnace heated to 1 000°C for 15-20 minutes.

10.3.2 Cool, place the crucible in a 500-ml beaker, add 100 ml water and 20 ml concentrated hydrochloric acid and heat to dissolve the melt. Wash the crucible with hot water and remove from the beaker.

10.3.3 Add 50 ml water and some filter paper pulp and filter through Whatman No. 41 filter paper and wash with hot water.

10.3.4 Dilute the filtrate and washings to 250 ml, heat to boil. Neutralise the excess acid with dilute ammonium hydroxide solution using methyl red indicator. Acidify the solution again by adding 2-3 ml of concentrated hydrochloric acid. Add slowly 10 ml of barium chloride solution and stir. Allow to stand for overnight. Filter through Whatman No. 42 filter paper and wash well with the hot water till free from chloride. Ignite the precipitate in a weighed platinum crucible at 600°C and then finally at 800°C. Cool in a desiccator and weigh the residue to the content weight.

10.3.5 Blank — Carry out a reagent blank using the procedure given above without the sample.

10.4 Calculation — Calculate the percentage of sulphate as follows:

$$\text{Sulphate (as } \text{SO}_3 \text{), percent by mass} = \frac{(A - B)}{C} \times 34·30$$

where

A = mass in g of barium sulphate obtained in the sample,

B = mass in g of barium sulphate obtained in the blank, and

C = mass in g of sample taken.

11. DETERMINATION OF PHOSPHOROUS

11.1 Outline of the Method — Sample is fused with sodium carbonate and boric acid and fused mass is treated with water. The sample solution is adjusted to *pH* 1·6 to 2·0 and treated with ammonium molybdate to form a complex and reduced with stannous chloride and measured at 710 nm in a spectrophotometer.

11.2 Reagents

11.2.1 Sodium Carbonate, Anhydrous

11.2.2 Boric Acid

11.2.3 Dilute Sulphuric Acid — 1 : 3 (v/v).

11.2.4 Hydroxyl Amine Hydrochloride — 2 percent.

11.2.5 Ammonium Molybdate Solution — Dissolve 3 g of ammonium molybdate in 60 ml concentrated hydrochloric acid and make up to 100 ml with distilled water.

11.2.6 Stannous Chloride (Stock Solution) — Dissolve 10 g of stannous chloride in 25 ml of concentrated hydrochloric acid.

11.2.6.1 Stannous chloride (working solution) — Dilute 1.5 ml of stock solution to 250 ml with distilled water.

11.2.7 Standard Phosphate Solution — Weigh 0.1917 g of dried potassium dihydrogen phosphate (KH_2PO_4), transfer to a 1000-ml volumetric flask and make up to the mark (1 ml = 0.1 mg P_2O_5).

11.2.7.1 Dilute 10 ml of the above solution to 100 ml in a volumetric flask (1 ml = 0.01 mg P_2O_5).

11.3 Procedure

11.3.1 Weigh 1 g of sample, mix with 2 g of boric acid and 4 g of sodium carbonate in a platinum crucible. Incinerate the contents from low bunsen flame to muffle furnace. Swirl the crucible and contents and heat again for 10 minutes. Remove the crucible from the furnace and cool. Fill the crucible with water. Heat gently until the entire melt has dissolved, then cool the solution and transfer to a 250-ml beaker containing about 100 ml of water. Rinse the crucible with 10-20 drops of dilute sulphuric acid and transfer to the beaker with few millilitres of water. Adjust to pH 1.6 to 2.0 with dilute sulphuric acid.

11.3.1.1 Add 5 ml of hydroxylamine hydrochloride solution and add 10 ml of ammonium molybdate solution. After 10 minutes add 5 ml of stannous chloride solution (working solution). Transfer the contents to 250-ml volumetric flask. After 10 minutes measure the absorbance of the solution against a reagent blank in a spectrophotometer at 710 nm.

11.3.2 Blank — Weigh and mix 2 g of boric acid and 4 g of sodium carbonate. As described in the sample fusion, dissolve the melt, transfer to a 250-ml beaker containing about 100 ml of water and adjust pH 1.6 to 2.0. Proceed in accordance with **11.3.1.1**.

11.3.3 Calibration Curve — Transfer 0, 2, 4, 6, 10, 15 and 20 ml of phosphate solution in seven 250-ml beakers. Proceed in accordance with **11.3.1.1** and plot the photometric readings of calibration solution against mg of $\text{P}_2\text{O}_5/250$ ml solution.

11.3.4 Calculation — Convert the photometric readings of sample and blank solution to mg of phosphate by means of calibration curve and calculate the percentage of phosphorous (as P_2O_5) as follows:

$$\text{Phosphorus (as } P_2O_5 \text{)} = \frac{A - B}{C} \times \frac{1}{10}$$

where

A = mass in mg of P_2O_5 found in sample,

B = mass in mg of P_2O_5 found in blank, and

C = mass in g of sample taken for the test.

(SMDC 35)

Indian Standard

METHODS OF

CHEMICAL ANALYSIS OF

ALUMINIUM TRIFLUORIDE

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF ALUMINIUM TRIFLUORIDE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 April 1967, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Aluminium trifluoride is mainly used in the aluminium industry for the production of aluminium from alumina. This standard prescribes methods for the correct analysis of aluminium trifluoride which may primarily be used as referee methods and may also be found useful for routine analysis.

0.3 In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by deriving assistance from the '1964 Book of ASTM Methods for chemical analysis of metals: Part 32' issued by the American Society for Testing and Materials.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard covers methods of chemical analysis for fluorine and other constituents of aluminium trifluoride general limits for which are given below:

<i>Constituent</i>	<i>Percent</i>
Total water	0.5 <i>Max</i>
Fluorine as aluminium fluoride	85 <i>Min</i>
Silica	0.5 <i>Max</i>
Iron oxide	0.3 <i>Max</i>

*Rules for rounding off numerical values (revised).

2. SAMPLING

2.1 The laboratory sample shall be prepared in accordance with methods prescribed in IS : 3191*.

3. GENERAL

3.1 **Use of Filter Papers**— In the methods prescribed in this standard, only relative numbers of Whatman filter papers have been prescribed since they are commonly used. However, any other suitable brand of filter paper of corresponding porosity may also be used.

4. QUALITY OF REAGENTS

4.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1960†) shall be employed in the tests.

NOTE— 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

5. DETERMINATION OF TOTAL WATER BY THE ABSORPTION METHOD

5.1 **Outline of the Method**— The total water is separated by heating the sample in a jet of dry air, and is collected into absorption tube containing magnesium perchlorate.

5.2 **Apparatus**— The apparatus as assembled in Fig. 1 consists of the following units:

- a) **Air Drying Unit**— It consists of a tower (A) packed with potash sticks, a tower (B) containing magnesium perchlorate. The main function of the unit is to deliver dry air to the combustion tube (C).
- b) **Combustion Tube (C)**— It consists of a porcelain tube, 500 to 600 mm in length with an inner diameter of 18 to 20 mm; one end of the combustion tube, containing lead oxides ($PbO + PbO_2$) and pumice in equal proportion is connected to the water absorption unit and the other end is connected to the air drying unit.
- c) **Combustion Furnace (D)**— The combustion furnace is an electric furnace, having two heating sections and capable of maintaining the first part of the tube between 800° and 900°C by means of (D_1). The other section (D_2) heats the end of the combustion tube, containing lead oxides and pumice between 250° and 300°C.
- d) **Water Absorption Unit**— It consists of two U tubes (U_1) and (U_2) packed with magnesium perchlorate. Each absorption tube weighs not more than 40 g.
- e) **Sulphuric Acid Bubbler (E)**— filled with concentrated sulphuric acid (see 5.3.2).

*Methods of sampling cryolite and aluminium trifluoride (under preparation).

†Specification for water, distilled quality (revised).

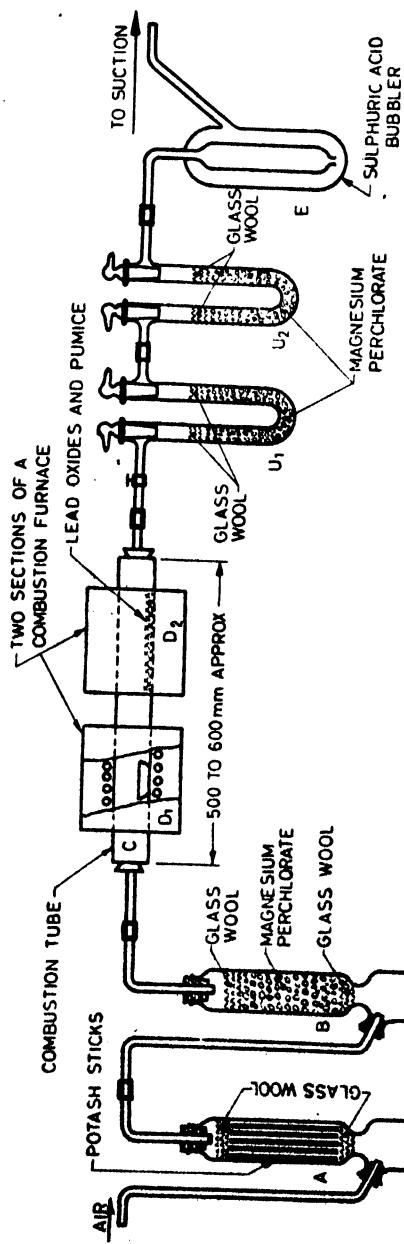


FIG. 1 APPARATUS FOR DETERMINATION OF TOTAL WATER

5.3 Reagents

5.3.1 *Dilute Hydrochloric Acid* — 1 : 1 (v/v).

5.3.2 *Concentrated Sulphuric Acid* — sp gr 1.84 (conforming to IS : 266-1961*).

5.3.3 *Magnesium Perchlorate* — solid.

5.3.4 *Lead Oxide* — solid.

5.3.5 *Lead Peroxide* — solid.

5.4 Procedure

5.4.1 Check the assembled apparatus for hermetic sealing. Before beginning the test, allow the air to flow through U-tubes at a rate of two to three bubbles per second. Simultaneously, switch on the combustion furnace and heat the first section (D_1) to 800° to 900°C. Bring the temperature of the section (D_2) of the furnace, heating a part of the tube containing the lead oxides and pumice, to 250°C. Connect the U-tubes after 30 minutes with the apparatus and pass air for half an hour. Then disconnect the tubes and weigh, after equalizing the pressure by opening the stopcocks of the tubes for a moment.

5.4.2 Reconnect the U-tubes and suck in the air through the apparatus for 30 minutes maintaining the specified temperatures, after which reweigh the tubes. Repeat the procedure, if necessary, until the weights of the tube (U_1) and (U_2) are constant.

5.4.3 Cool down the combustion tube to about 120°C and introduce a platinum or porcelain boat containing about one gram of accurately weighed sample. Connect the absorption tubes and suck in dry air through the apparatus for five minutes at the rate of two bubbles per second. Simultaneously, switch on the furnace, bring the heating section (D_1) to temperature range between 800° to 900°C, and heating section (D_2) between 250° to 300°C. Maintain the temperature for 30 minutes. Stop heating without stopping the flow of dry air and disconnect the absorption tubes (U_1) and (U_2), closing their stopcocks immediately. Allow the tubes to cool to room temperature and weigh after equalizing the pressure by opening the stopcocks of the tubes momentarily.

5.5 Calculation

$$\text{Total water, percent} = \frac{(A+B)}{C} \times 100$$

where

A = increase in weight in g of U-tube (U_1),

B = increase in weight in g of U-tube (U_2), and

C = weight in g of the sample taken.

*Specification for sulphuric acid (revised).

6. DETERMINATION OF FLUORINE BY DISTILLATION METHOD

6.1 Outline of the Method — Fluorine is steam distilled from the solution of the sample containing perchloric acid and titrated with standard thorium nitrate solution.

6.2 Apparatus — The distillation apparatus (*see* Fig. 2) consisting of a 250-ml distillation flask (*A*), side arm of which is connected to a condenser (*B*). The neck of the flask is fitted with a two-holed rubber stopper carrying a thermometer (*C*) and 4-mm glass tube (*D*) connecting the flask to a steam generator (*E*). The latter is equipped with two discharges, one tube (*F*) to release steam and the other tube (*D*) to deliver steam into the distillation flask (*A*) and a safety tube about 90 cm. The distillate is collected in a conical flask (*G*).

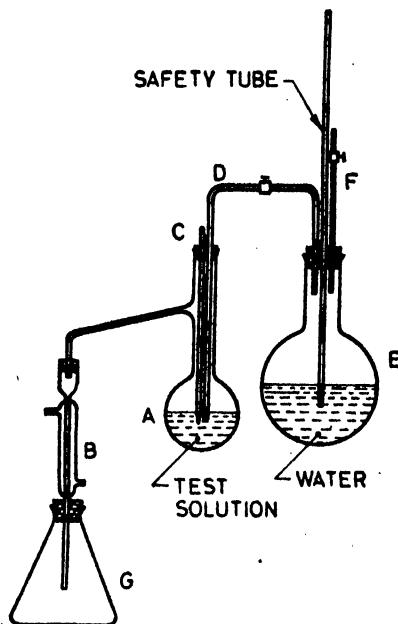


FIG. 2 APPARATUS FOR DISTILLATION OF FLUORINE

6.3 Reagents

6.3.1 Sodium Carbonate — solid.

6.3.2 Potassium Carbonate — solid.

6.3.3 Ignited Silica or Finely Ground Quartz

6.3.4 Perchloric Acid — 70 percent.

6.3.5 Sodium Alizarin Sulphonate Indicator — Dissolve 0.050 g of sodium alizarin sulphonate in 100 ml of water and store in a dropping bottle.

6.3.6 Sodium Hydroxide Solution — Dissolve 20 g of sodium hydroxide pellets in water, cool and dilute to one litre. Mix well.

6.3.7 Dilute Hydrochloric Acid — Dilute 19.6 ml of concentrated hydrochloric acid (sp gr 1.16) to 1 000 ml with water. Mix well.

6.3.8 Buffer Solution — Dissolve 9.448 g of monochloracetic acid in 100 ml of water. Add 50 ml of sodium hydroxide (1.0 N). Mix well.

6.3.9 Standard Thorium Nitrate Solution (0.025 M) — Weigh 13.806 g of thorium nitrate $[\text{Th}(\text{NO}_3)_4 \cdot 4\text{H}_2\text{O}]$; dissolve in water and make up to one litre. Mix well. Standardize against standard sodium fluoride as under 6.4.3.

6.3.10 Standard Sodium Fluoride Solution (1 ml = 1 mg of F) — Dissolve 2.211 g of sodium fluoride (AR), previously dried at 120°C to constant weight, in one litre of water in a volumetric flask.

6.4 Procedure

6.4.1 Weigh accurately 0.25 g of the sample and fuse it with 1.1 g of sodium carbonate, 1.4 g of potassium carbonate and 0.6 g of ignited silica, or finely ground quartz in a platinum crucible. Transfer the melt with the aid of little water to 250-ml distillation flask (A) containing 8 to 10 glass beads. Add 30 ml of perchloric acid. The temperature should not rise above 50°C.

6.4.2 Place a conical flask (G) under the condenser. Connect the apparatus as shown in Fig. 2. Distil the solution until the boiling point reaches about 140°C. While this is being done, heat the water in the steam generator (E) to boiling but do not allow the steam to get in the flask (A). When the temperature reaches 165°C, admit steam into the flask (A). Maintain the temperature at about 140°C by adjusting the steam flow and heating the flask (A). Distil at such a rate that no fumes or mist escape from the flask (G). Continue distillation until nearly 200 ml are collected in a conical flask (G). Transfer the distillate to a 250-ml volumetric flask.

6.4.3 Cool and make up the volume to the mark and mix well. Pipette out 50 ml aliquot into a 250-ml beaker. Add 50 ml of water, 10 drops of sodium Alizarin-sulphonate indicator and mix. Add sodium hydroxide solution slowly with stirring till the solution becomes pink. Add dilute hydrochloric acid drop by drop with stirring till the pink colour is just discharged leaving the solution just acid. Add from a burette 2 ml of buffer solution and titrate with standard thorium nitrate solution with constant

stirring, till a permanent pink end point is reached. The colour intensity at the end point should be kept in mind and the titration should be stopped exactly at the same colour intensity for both the thorium nitrate standardization and the test solution titration.

6.5 Calculation

$$\text{Fluorine, percent, } D = \frac{A \times B}{C} \times 100$$

where

A = volume in ml of standard thorium nitrate solution,

B = fluorine equivalent of the standard thorium nitrate solution in g/ml, and

C = weight in g of the sample represented by the aliquot taken.

6.5.1 Aluminium trifluoride, percent = $D \times 1.473$.

7. DETERMINATION OF SILICA BY THE MOLYBDENUM BLUE (PHOTOMETRIC) METHOD

7.1 Outline of the Method — The sample is decomposed with fusion mixture and acidified with nitric acid. The orthosilicic acid formed is converted to yellow silicomolybdic acid by the addition of ammonium molybdate to the solution. The complex is reduced to blue silicomolybdic acid and photometric measurement is then made at about $810 \text{ m}\mu$.

7.2 Reagents

7.2.1 Fusion Mixture — Mix thoroughly 250 g of anhydrous sodium carbonate with 100 g of boric acid.

7.2.2 Aluminium Chloride Solution — Dissolve exactly 200 g of aluminium chloride ($\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$) in 800 ml of water containing small amount of hydrochloric acid. Transfer to a one-litre flask. Dilute to the mark with water and mix well.

7.2.3 Standard Nitric Acid — Dilute 540 ml of nitric acid (sp gr 1.42) to 1000 ml with water. Check the strength of the solution by titrating 10.600 g of anhydrous sodium carbonate (Na_2CO_3) previously dried in a hot air oven (at approximately 110°C) and using methyl red (0.02 percent in alcohol) as indicator. The theoretical quantity of nitric acid for the above quantity of sodium carbonate is 25 ml. If necessary, adjust the concentration of the nitric acid solution by adding water or acid so that the solution corresponds to the strength of 8N.

7.2.4 Ammonium Molybdate Solution (10 Percent) (w/v) — Dissolve 100 g of ammonium molybdate [$(\text{NH}_4)_6\text{MO}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$] in water and dilute to one litre. Filter before use.

7.2.5 Standard Sulphuric Acid— Dilute 225 ml of sulphuric acid (sp gr 1.84) to 1 000 ml with water. Check the strength by titrating it with standard sodium hydroxide solution (8N) and if necessary correct the concentration so that 10.0 ml of the sulphuric acid solution corresponds to 10.0 ± 0.1 ml of the standard sodium hydroxide solution (8N). Standardize sodium hydroxide against standard potassium acid phthalate solution.

7.2.6 Reducing Solution— As in 7.2.6.1 or 7.2.6.2 may be used.

7.2.6.1 Solution of 1-amino 2-naphthol 4-sulphonic acid— Dissolve 7 g of anhydrous sodium sulphite (Na_2SO_3) in 100 ml of water, then add 1.5 g of 1-amino 2-naphthol 4-sulphonic acid and stir the solution until dissolution is complete. Dissolve separately 90 g of sodium metabisulphite ($\text{Na}_2\text{S}_2\text{O}_5$) in approximately 800 ml of water. Mix the two solutions, filter through a medium texture filter collecting the filtrate and the washings in a 1 000-ml volumetric flask. Make up to the mark with water, mix and transfer the solution to a flask of yellow glass. Use a freshly prepared solution.

7.2.6.2 Ascorbic acid solution (20 g per litre)— Dissolve 20 g of ascorbic acid in water and make up the volume to 1 000 ml. Use a freshly prepared solution.

7.2.7 Sodium Fluoride— solid.

7.2.8 Aluminium Chloride ($\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$)— solid.

7.2.9 Standard Silica Solution (1 ml of = 0.04 mg of SiO_2)— Fuse 0.04 g of anhydrous silica with 1.0 g of sodium carbonate in a platinum crucible. Cool the melt, dissolve completely in water, and dilute to one litre in a volumetric flask. Store the solution in a polythene bottle. In case anhydrous silica is not available, adjust the weight of silica used according to the actual content of the sample.

7.3 Procedure

7.3.1 Transfer 0.25 g of an accurately weighed sample to a clean 40-ml platinum crucible containing 3.5 g of fusion mixture. Mix intimately by means of a platinum rod. Remove the rod, brush into the crucible any adhering solid and cover with platinum lid. Heat with Meker or other high-temperature burner gently at first, then gradually to the full strength of the burner. Continue heating, as strongly as possible, for 5 to 10 minutes.

7.3.2 Cool the crucible, dissolve the melt in water, filling the crucible as much as possible without risk of a loss. Heat gently without boiling, until the soluble salts are dissolved. Cool somewhat and transfer to a beaker containing 6.5 ml of standard nitric acid and 25 ml of aluminium chloride solution. Wash the crucible three or four times with hot water, and transfer the washings to the beaker. Mix by swirling, cover, heat to

incipient boiling, and then keep warm, without boiling, until a clear solution is obtained. Cool, transfer to a 250-ml volumetric flask, dilute the solution to the mark with water and mix well. Cool to room temperature (lower than 30°C).

7.3.3 Transfer 50 ml of the sample solution to a 100-ml volumetric flask. Add 0.9 ml of standard nitric acid. Dilute to 75 ml with water and mix. Add 5 ml of ammonium molybdate solution, mix and allow to stand for 5 minutes. Add 10 ml of standard sulphuric acid and 5 ml of reducing solution. Dilute the solution to the mark and mix well. Allow the solution to stand for ten minutes for full development of colour.

7.3.4 Reference Solution — Transfer 70 ml of water to 100-ml volumetric flask and proceed as under **7.3.3**.

7.3.5 Transfer a suitable portion of the reference solution to an absorption cell and adjust the photometer to the initial setting using a light filter at approximately 810 m μ . While maintaining this photometer adjustment, take the photometric readings within ten minutes after the full development of colour.

7.3.6 Reagent Blank — Make a blank determination, following the same procedure and using the same amount of reagents.

7.3.7 Calibration Curve

7.3.7.1 Transfer 7 g of fusion mixture to a 250-ml plastic beaker. Dissolve in 150 ml of hot water. Add from a burette 20.8 ml of standard nitric acid. In another plastic beaker dissolve 0.5 g of sodium fluoride in 100 ml of hot water. When sodium fluoride is dissolved, add 10 g of aluminium chloride and stir the solution. Transfer the two solutions to a 500-ml volumetric flask, cool, dilute to the mark and mix well. Store in a polythene bottle.

7.3.7.2 To a series of 100-ml volumetric flasks, transfer 50-ml aliquots of the above solution. Add measured amount of standard silica solution covering the range of silica and proceed as under **7.3.2**. Take photometer readings of the calibration solutions by following the same procedure as under **7.3.5**. Plot the photometer readings of the calibration solutions against milligrams of silica per 100 ml of solution.

7.4 Calculation — Convert the photometric readings of the sample and the reagent blank to milligrams of silica by means of calibration curve, and calculate the percentage of silica as follows:

$$\text{Silica, percent} = \frac{A - B}{C} \times \frac{1}{10}$$

where

A = weight in mg of silica found in the aliquot,

B = reagent blank correction, in mg of silica, and

C = weight in g of the sample represented by the aliquot taken.

8. DETERMINATION OF IRON OXIDE BY THE O-PHENANTHROLINE (PHOTOMETRIC) METHOD

8.1 Outline of the Method — The sample is decomposed by fusion with potassium bisulphate. From the melt dissolved in water, iron is determined photometrically as the orange-red complex that is formed between O-phenanthroline and ferrous iron. Measurement is made at approximately 490 $\text{m}\mu$.

8.2 Reagents

8.2.1 Potassium Bisulphate — anhydrous.

8.2.2 Concentrated Hydrochloric Acid — sp gr 1.16 (conforming to IS : 265-1962*).

8.2.3 Hydroxylamine Hydrochloride Solution — 10 percent (w/v).

8.2.4 2 : 4 Dinitrophenol Indicator — 0.1 percent (w/v).

8.2.5 Tartaric Acid — 10 percent (w/v).

8.2.6 Ammonium Hydroxide Solution — Mix together 120 ml of water and 80 ml of concentrated ammonium hydroxide.

8.2.7 Dilute Hydrochloric Acid (6 N) — Dilute concentrated hydrochloric acid with an equal volume of water.

8.2.8 O-Phenanthroline Solution — Dissolve 0.2 g of ortho-phenanthroline monohydrate in little ethanol and dilute to 100 ml with water.

8.2.9 Standard Iron Solution (1 ml = 0.02 mg of Fe) — Dissolve 0.1405 g of ferrous ammonium sulphate $[\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2, 6\text{H}_2\text{O}]$ in 100 ml of dilute hydrochloric acid (1:19) and dilute to one litre in a volumetric flask. Standardize against standard potassium dichromate solution. Prepare fresh as needed.

8.3 Procedure

8.3.1 Fuse one gram of sample with 20.0 g of potassium bisulphate in a 40-ml platinum crucible. Transfer the solidified melt to a 250-ml beaker. Dissolve any melt left in the crucible in hot water and add it to the beaker. Dilute to about 75 ml with water, add 5 ml of concentrated hydrochloric acid and heat to dissolve all soluble salts. When completely dissolved, transfer to a 100-ml volumetric flask, cool, dilute to the mark with water and mix well.

8.3.2 Pipette a suitable aliquot into a 100-ml beaker. Add 1 ml of hydroxylamine hydrochloride solution and mix.

*Specification for hydrochloric acid (revised).

8.3.3 Add 10 ml of tartaric acid. Add 0.5 ml of 2:4 dinitrophenol indicator to the solution. Add ammonium hydroxide solution dropwise with constant stirring until the solution assumes a slight yellow colour. Add dilute hydrochloric acid dropwise and with constant stirring until the yellow colour is just discharged.

8.3.4 Add 5 ml of O-phenanthroline solution. Stir well and transfer to a 100-ml measuring flask. Make up the volume with water. Mix well and allow to stand for about 30 minutes.

8.3.5 Reference Solution — Transfer 60 ml of water to 100-ml volumetric flask and proceed as under **8.3.2** to **8.3.4**.

8.3.6 Transfer a suitable portion of the reference solution to an absorption cell and adjust the photometer to the initial setting, using a light filter at 490 m μ . While maintaining this photometer adjustment, take photometer readings of the sample solution.

8.3.7 Reagent Blank — Make a blank determination, following the same procedure and using the same amounts of reagents.

8.3.8 Calibration Curve — Transfer 1.0, 2.0, 4.0, 8.0, and 12.0 ml of standard iron solution (see **8.2.9**) to five 100-ml volumetric flasks. Dilute to 60 ml and proceed in accordance with **8.3.2** to **8.3.4**. Take photometric readings of the calibration solutions by following the same procedure as under **8.3.6**. Plot the photometric readings of the calibration solutions against milligrams of iron per 100 ml of solution.

8.4 Calculation — Convert the photometric readings of the sample and blank solution to milligrams of iron by means of calibration curve and calculate the percentage of iron as follows:

$$8.4.1 \text{ Iron, percent} = \frac{A - B}{C}$$

where

A = weight in mg of iron found in the aliquot,

B = reagent blank correction in mg of iron, and

C = weight in g of the sample represented by the aliquot taken.

$$8.4.2 \text{ Iron oxide (Fe}_2\text{O}_3\text{), percent} = \text{iron percent} \times 1.4298.$$

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